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TENSILE PROPERTIES OF 20% COLD-WORKED TITANIUM-MODIFIED TYPE 316 STAINLESS STEEL IRRADIATED IN HFIR*

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An alloy of type 316 stainless steel with the addition of 0.23% Ti (316 + Ti) has been irradiated in the 20% cold-worked condition in the HFIR (a mixed fast and thermal neutron spectrum reactor) and tested near the irradiation temperature in the range of 300–600°C. Tensile tests were performed following irradiation to fluence levels of 0.63–2.1 × 10^{26} n/m^2 (E > 0.1 MeV) and helium levels of 200–1000 at. ppm. The 316 + Ti exhibited higher strength and lower ductility than similarly irradiated type 316 stainless steel (316 SS). However, the tensile elongation of 316 + Ti tends to saturate with increasing fluence at 575°C whereas the elongation of 316 SS continues to fall for the fluences investigated. Reduction of area is similar for the two alloys, and 316 + Ti shows completely ductile rupture at 450°C and below. The differences in strength and ductility are attributed to the influence of TiC precipitates trapping helium in the matrix.

1. INTRODUCTION

A fusion reactor first wall requires a material to sustain a high flux of alpha particles, radiant energy, and high energy neutrons. It is the high energy (14.1 MeV) neutrons that produce radiation damage throughout the bulk of the structure that are of concern to us in this research. Type AISI 316 stainless steel (316 SS) has been selected as a candidate alloy for consideration in the United States Fusion Energy Program because of its radiation performance and because of the large body of data available on the alloy from the Breeder Reactor program. Tensile properties of 20%-cold-worked type 316 stainless steel (20%-CW 316 SS) irradiated in the High Flux Isotope Reactor (HFIR) have been reported previously [1]. However, significant improvements have been obtained in this alloy in both high-temperature mechanical properties and in irradiation-induced swelling resistance by small additions of titanium [2,3]. The present study is focused on type 316 stainless steel to which has been added 0.23 wt % Ti (referred to as 316 + Ti). The composition is shown in Table 1. This material was irradiated in the HFIR simultaneously and under the same conditions as the standard 20% CW 316 SS, the mechanical and fracture properties of which are described in ref. [1].

Exposure of most common structural materials to neutrons above 10 MeV results in the formation of helium through (n,α) reactions. Since helium is known to have detrimental effects on mechanical properties [4], materials being developed for fusion irradiation resistance should be irradiated under conditions that simultaneously produce atom displacement damage and helium. No 14 MeV neutron sources that provide sufficiently high flux for tensile property studies are available; however, mixed-spectrum (fast + thermal) fission reactors will impart both displacement damage and helium to nickel-bearing alloys such as stainless steels. The helium is formed primarily by a two-step thermal neutron absorption reaction sequence beginning with ^{58}Ni [5]. The helium to dpa ratios obtained in austenitic stainless steels in HFIR are significantly higher than would result from fusion reactor irradiation. However, comparison of ductility and fracture properties of alloys with high helium contents remains of interest. Strength, which depends largely upon the defect structure produced by displacement damage, saturates at about 10 dpa and is fairly insensitive to helium level. The loss of high-temperature ductility is dominated by such things as helium since strength has saturated. Therefore, above 10 dpa the ratio of He:dpa is expected to be less important than the helium content itself for qualitative comparison of one alloy with another.

Table 1. Composition of (316 + Ti) SS

	Content (wt %)		Content (wt %)
Cr	17.0	C	0.06
Ni	12.0	P	0.01
Mo	2.50	S	0.013
Mn	0.5	N	0.0055
Ti	0.23	B	0.0007
Si	0.40	Fe	Bal

2. EXPERIMENTAL PROCEDURES

Miniature tensile specimens with a gage length of 18.3 mm and a gage diameter of 2.03 mm were fabricated from 316 + 0.23 wt % Ti. The specimens were irradiated in HFIR in a peripheral target position, where the peak thermal flux is 2.5 × 10^{19} n/m^2·s and the peak fast flux is 1.3 × 10^{19} n/m^2·s (>0.1 MeV). The specimen holder is of the same design as that used by Bloom and Wiffen [6]. A gas gap was provided around each specimen to restrict radial heat transfer to obtain the desired temperature.

Specimen material was initially annealed 1 h at 1150°C then swaged and annealed to obtain the

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proper diameter with a final anneal of 1 h at 1050°C. The material was then swaged to 20% reduction in area.

No instrumentation was installed in the irradiation capsule; however, extensive dosimetry has been done in the past, and temperature monitors from more recent irradiations are now being analyzed. Preliminary analysis of temperature monitors indicates that irradiation temperatures may have been higher than calculated by a maximum of 50–75°C. However, the calculated temperatures are consistent with previously reported data, and the comparison between 316 SS and 316 + Ti remains valid. Helium levels were calculated from an empirical relation determined by Wiffen [7] and based on mass spectrographic analysis of HFIR-irradiated specimens. The calculations are believed to have an accuracy of ±20%.

Tensile tests were conducted on an Instron tensile testing machine using a nominal strain rate of 4.6×10^{-5} s^{-1}. Tests were performed in air in a resistance furnace with a 150-mm hot zone to provide temperature uniformity over the center region of 50 mm. Test temperatures were selected close to calculated irradiation temperatures, and specimens tested at 300, 350, 450, 575 and 600°C were chosen for more detailed data analysis. Fluences ranged from 0.63 to 2.1×10^{26} n/m^2 (>0.1 MeV), displacement damage levels from 5 to 16 dpa, and helium contents from 200 to 1000 at. ppm.

Following tensile testing, reduction of area measurements were made. This was accomplished by making a low-magnification fractograph in a scanning electron microscope (SEM). The fracture surface area was measured and reduction of area computed using a standard for magnification calibration for each specimen. The true fracture stress was obtained from the load and area at fracture.

3. RESULTS

The results of the tensile tests appear in Table 2. Only yield strength and total elongation at all temperatures and reduction of area at 350, 450, and 575°C have been selected to be plotted graphically in Figs. 1–3. Where possible, similar data on type 316 stainless steel irradiated in the same series of irradiation capsules as the 316 + Ti alloy have been plotted for comparison [1]. As in the case of the 316 SS, where the variables plotted change rapidly, straight-line segments were drawn between points. The 0.2% yield stress (Fig. 1) increases with increasing fluence at 300 and remains constant at 350°C, but decreases at 450, 575, and 600°C with the strength at 600°C nearly equal to that at 575°C. Yield

Table 2. Tensile Properties of 20% CW 316 + Ti Stainless Steel Irradiated in HFIR

Sample	Temperature (°C)		Neutron Fluence >0.1 MeV ($n/m^2 \times 10^{26}$)	dpa	Helium Content (at. ppm)	Strength (MPa)		Elongation (%)		Reduction of Area (%)	True Fracture Stress (MPa)
	Test	Irradiation				Yield	Ultimate	Uniform	Total		
AD01	300		0			772	821	0.72	5.9	54	1125
F7	300	285	1.0	7.7	390	903	903	0.19	5.8	68	1548
F27	350		0			759	779	0.56	5.7	64	
F29	350		0			786	814	0.87	5.9	61	1320
F13	350	370	0.63	4.9	180	752	786	3.1	8.5	66	1292
F15	350	375	1.1	8.5	380	786	848	2.6	7.0	58	1289
F2	350	375	1.7	13	740	772	855	4.2	8.4	55	1147
F10	350	375	1.7	13	740	758	834	4.7	9.0	54	1219
AD03	450		0			754	798	0.86	6.0	55	1195
F26	450		0			710	765	2.1	6.9	66	1351
F22	450	465	0.9	6.9	290	643	717	3.7	7.6	51	941
F21	450	475	1.3	10	500	603	696	3.7	7.2	30	747
F6	450	475	2.1	16	980	588	687	4.7	8.3	40	856
F23	575		0			643	687	1.3	5.8	67	1336
F28	575		0			678	731	2.0	6.8	54	
F14	575	560	0.9	6.9	290	490	581	4.5	7.2	45	908
F16	575	565	1.2	9.2	440	462	570	4.1	4.6	14	608
F1	575	560	1.4	11	600	519	600	3.9	4.4	12	599
F11	575	565	1.9	15	880	400	497	3.1	3.5	17	560
AD10	600		0			572	585	0.94	6.4	53	952
F20	600	620	1.3	10	530	430	528	2.7	3.0	21	601
F5	600	620	2.1	16	1020	395	495	2.6	3.1	62	507

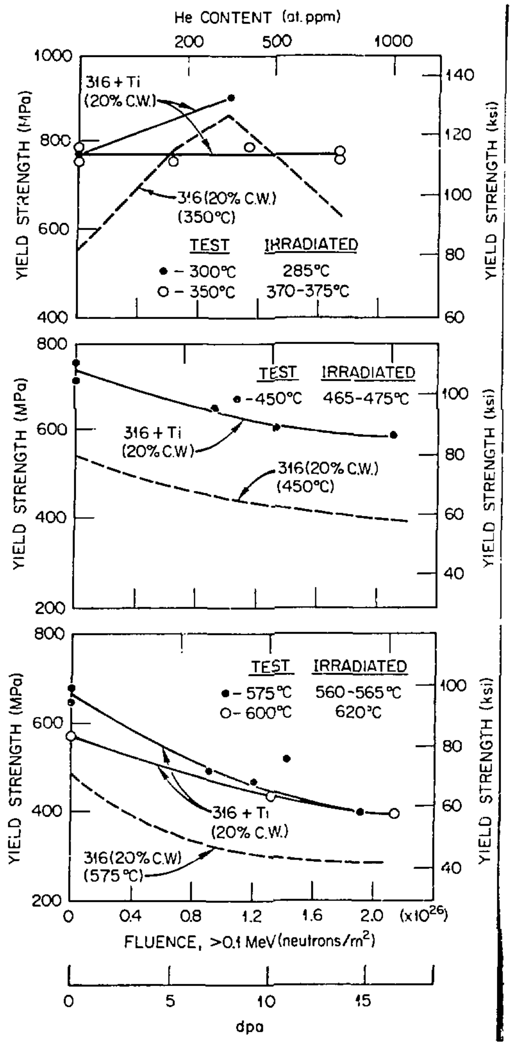

Figure 1. Yield strength versus fluence for 20% CW 316 + 0.23 Ti irradiated in HFIR. Dashed curves are for 20% CW 316.

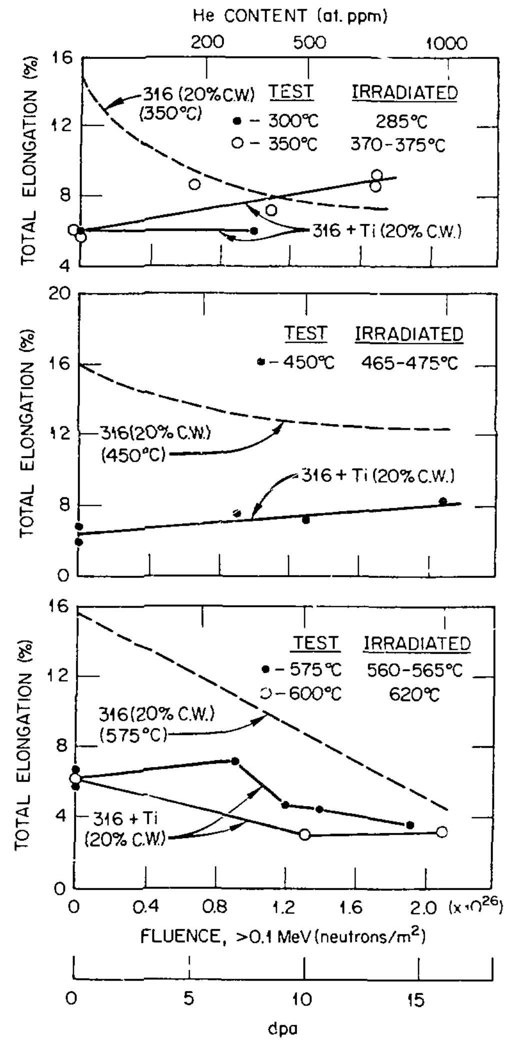

Fig. 2. Total elongation versus fluence for 20% CW 316 + 0.23 Ti irradiated in HFIR. Dashed curves are for 20% CW 316.

strength appears to saturate by a fluence of 2.0×10^{26} n/m^2 at all temperatures where sufficient data exist to draw a conclusion. The nearly constant yield strength at 350°C for the 316 + Ti is to be contrasted with a rather sharp maximum in the strength of 316 SS which results from a change in precipitation behavior (Fig. 1) [8]. At 450 and 575°C the variation in strength with fluence is similar to that of 316 SS, and both decline with increasing fluence with 316 + Ti 30–50% stronger.

Tensile elongation appears in Fig. 2 where the ductility of 316 + Ti can be seen to be significantly lower than that of 316 SS initially. However, at 350 and 450°C the ductility of 316 + Ti is nearly constant or slightly increasing with fluence and does not exhibit the initial drop in ductility characteristic of 316 SS. Only at 450°C does the total elongation of 316 SS remain significantly greater than that of 316 + Ti, but both appear to remain above 8%. At 575°C the total elongation of 316 + Ti and 316 approaches similar values at about 2×10^{26} n/m^2. However, the 316 + Ti appears to saturate above 3% elongation at 575°C and this is supported by the similar behavior at 600°C. By comparison, the total elongation of 316 is declining rapidly without any indication yet of saturation.

Although not plotted, the values of uniform elongation (Table 2) show an interesting trend. Unirradiated CW 316 + Ti tested at 350 and 450°C exhibits uniform elongation below 1% but after HFIR irradiation, this parameter is greater and increases with fluence. A similar but less dramatic effect persists at 575 and 600°C.

Reduction of area (RA) is more indicative of the fracture mechanism. Figure 3 is a plot of RA data for 316 + Ti with dashed curves for CW 316 with accompanying representative SEM fractographs. RA behavior for 316 + Ti is nearly the same as 316 SS. Of particular interest, RA appears to remain above 10% at 575°C similar to 316 SS. Even at 600°C RA is above 6% at 2.1×10^{26} n/m^2. The failure occurs by ductile rupture at 450°C and below, but intergranular separation is the primary mode at 575°C and above and is responsible for the rapid reduction in ductility with increasing fluence as the tendency toward intergranular fracture increases.

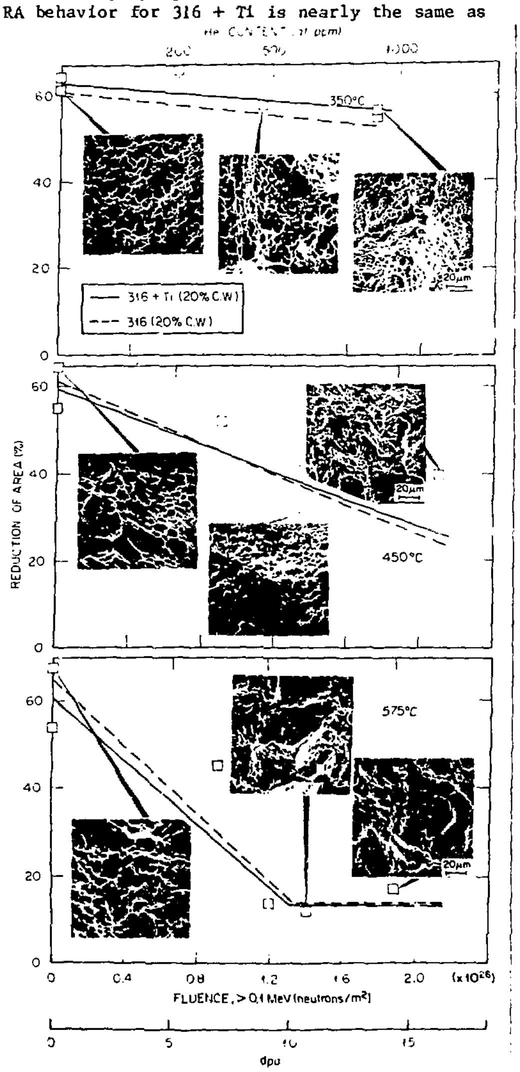

Figure 3 : Reduction of area versus fluence for 20% CW 316 + 0.23% Ti irradiated in HFIR. Representative fracture surfaces are shown. Dashed curves are for CW 316.

Transmission electron microscopy (TEM) was performed on material cut from the grip ends of the specimens following tensile testing. These have been shown to contain the as-irradiated microstructure with little or no recovery or deformation due to the elevated temperature testing. Detailed microscopy is reported elsewhere [8]. Three micrographs of specimens irradiated to approximately 1.3×10^{26} n/m^2 (10 dpa) appear in Fig. 4. The specimen tested at 350°C (F15) has a high density of Frank interstitial loops as well as a dislocation network. The dislocation density in the cold-worked material has recovered somewhat during irradiation but fine Ti-rich MC precipitate particles are pinning dislocations. Loops contribute ~25% to the total observed dislocation structure at 1.1×10^{26} n/m^2 and 350°C. The specimen irradiated at 475°C (F21) shows considerable recovery of dislocations and only slight coarsening of the still rather fine MC. The CW 316 specimen irradiated at 565°C (F16) shows greater recovery of the dislocation structure as well as further MC coarsening.

4. DISCUSSION

The electron micrographs in Fig. 4 are helpful in understanding the tensile properties. At 350°C the observed strengthening is attributed mostly to the fine MC pinning dislocations since yield strength remains insensitive to increasing fluence (Fig. 1) even though the loops have been shown previously to nearly vanish by a fluence of 1.7×10^{26} n/m^2 [8]. At 450–475°C dislocation recovery, MC coarsening and additional solute depletion by precipitation contribute to the decreased yield strength compared to 350°C for CW 316 + Ti. The increment of strength of 316 + Ti over that of 316 SS at 450°C is attributed to MC pinning plus a reduced loss of solutes through precipitation. The greater recovery and increased MC coarsening observed at 565–575°C both contribute to the lower strength at this temperature. Arguments similar to those for 450°C explain the strength difference between CW 316 and CW 316 + Ti at 575°C. Increased strength usually results in lower tensile elongation, consistent with the initial difference for 316 + Ti compared to 316 SS. The lower ductility at 575 and 600°C compared to lower temperatures for the same fluences is caused by the onset of intergranular fracture in both alloys [1]. This intergranular fracture occurs at lower temperatures and fluences than in similar material irradiated in EBR-II [9] and is attributed to the presence of significant grain boundary helium bubbles coincident with loss

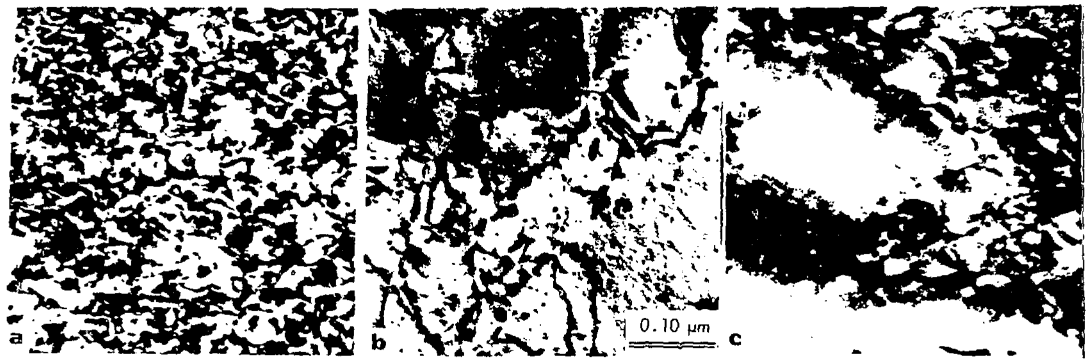

Figure 4 : Representative microstructures of 316 + 0.23 Ti. Irradiated in HFIR at: (a) 375°C, (b) 475°C, and (c) 565°C.

of the normal grain boundary precipitation (8). Cold-worked 316 + Ti is significantly stronger at 575°C and only slightly less ductile at 2×10^{26} n/m^2 than CW 316 SS. RA and fracture mode are similar and, importantly, the ductility is changing little with increasing fluence for CW 316 + Ti in contrast to CW 316 (Fig. 2). TEM observations have shown grain boundary cavities to be smaller in CW 316 + Ti than in CW 316 SS [8]. Helium embrittlement is occurring in both alloys, but this observation helps explain the slower reduction in ductility with fluence in the titanium-modified alloy. Grain boundary cavity growth appears to saturate with increasing fluence at a given temperature [8], consistent with an apparent saturation in ductility, particularly in CW 316 + Ti where cavity growth saturates earlier at a smaller size.

Low values of uniform elongation which increase with fluence have been observed in 20% cold-worked type 316 stainless steel [10]. This limited ability to work harden probably results from a high initial dislocation density. Partial recovery during irradiation, primarily due to absorption of irradiation-produced interstitials that force climb, is believed to be responsible for the increase in uniform elongation.

It is noteworthy that at 450°C, all observed failures for 316 + Ti were by ductile rupture. This was not the case for 316 SS irradiated under similar conditions where transgranular cleavage-like fracture similar to channel fracture was observed [1]. This fracture mechanism is believed to result from excessive precipitation of η phase during irradiation along slip bands resulting from the initial cold work in 316 SS. Since such precipitation is absent in 316 + Ti, this form of brittle fracture does not occur.

CONCLUSIONS

1. 20% CW 316 + 0.23 Ti is stronger than type 20% 316 SS before and after irradiation.
2. Tensile elongation is lower for 316 + 0.23 Ti, but is much less sensitive to irradiation than 316 SS at 575–600°C.
3. The higher strength and weaker dependence of ductility on fluence of 316 + Ti are attributed to several beneficial functions of MC precipitates.
4. Reduction of area of 316 + Ti and 316 SS are nearly equal in the fluence range investigated – 0.63–2.1 × 10^{26} n/m^2 (E > 0.1 MeV).
5. Fracture is by ductile rupture for temperatures of 450°C and below, but by intergranular failure for 575 and 600°C above a fluence of 1.2×10^{26} n/m^2 (E > 0.1 MeV). The tendency for intergranular fracture increases with increasing fluence.

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